A Modified Pinkerton-Type He Gas-Flow System for High-Accuracy Data Collection at the X3 SUNY Synchrotron Beamline at NSLS

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Introduction: Use of cryogenic temperatures is of crucial importance in accurate diffraction studies. In charge density analysis it reduces thermal smearing of the electron density and allows collection of much higher order data, which leads to improved resolution in the final results. In time-resolved studies the cooling is essential for heat dissipation from the laser-illuminated sample. For transition metal carbonyl and some transition metal nitrosyl complexes photoinduced species are only stable below the temperature of liquid nitrogen.

Results: In preceding years we have used a He Displex refrigerator to attain a temperature of 20K. However, slight instabilities in the position the heavy Displex unit in the horizontal configuration required at the synchrotron source, and the limited accessibility for exciting light beams required a different design. We have now implemented an open gas-flow system, which, with some modifications, follows the design of Pinkerton *et al.* The system is based on a modified ESR evaporator, which produces both the inner cold stream and a dry outer He stream. Temperature calibration is based on the well-documented temperature-dependent fluorescence properties of the dinuclear platinum complex $K_4Pt_2(H_2P_2O_5)_4$ (Markert *et al*. 1983), which we can monitor because of the spectroscopic equipment installed at X3A.

A typical He temperature result is the deformation density in the carboxyl plane of L-leucyl-L-alanine tetrahydrate (Görbitz, 1997) is shown in **Figure 1a**. Data were collected on a crystal of 180×80×50 μm using two different rotation axes. The resolution in the experimental deformation density map is remarkable, especially in the C-C bond region, which shows a double maximum, which is retained on modeling. Noise levels are satisfactorily low. Some extra detail is added in the 'static' model map (**Figure 1b**), indicating that thermal motion can never be completely eliminated, and perhaps that at this temperature a higher data cut-off would be desirable even for valence density features. The full study of L-leucyl-L-alanine tetrahydrate will be reported separately.

Conclusions: Open gas systems eliminate stability problems encountered when heavy cryostats are operated in a horizontal orientation at a horizontally polarized synchrotron beam (Volkov *et al.* 1999). The use of an open gas flow He system is now feasible at high intensity synchrotron sources equipped with area detectors, at which data collection times are minimized.

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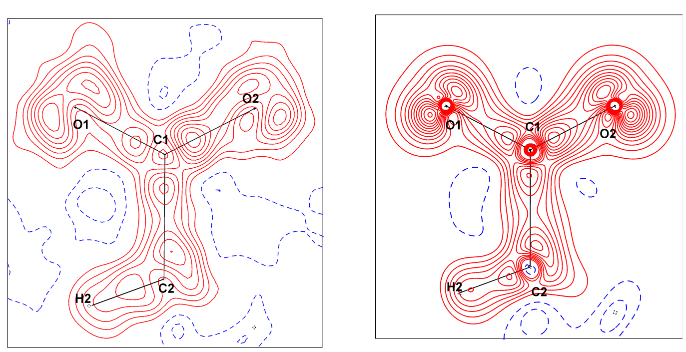

(a) (b)

Figure 1. Deformation density in the plane of the carboxylic group in L-Leucyl-L-alanine at 16K. (a) by Fourier summation of the experimental structure factors, reference parameters from multipole refinement. (b) model map based on multipole population parameters. Contours at 0.1 eÅ^{-3}. Negative contours broken. Zero contour omitted. Sinθ/λ_{max} 1.12 Å^{-1}, λ = 0.643 Å.